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FABRICATION OF GRAPHITE-ALUMINUM COMPOSITES VIA PULTRUSION.(U)

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DAA646-77-C-0036

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MARCH 1978

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FINAL REPORT,

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Contract Number DAAG46-77-C-0036

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Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AMMRC TR78-16	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Fabrication of Graphite-Aluminum Composites via Pultrusion		5. TYPE OF REPORT & PERIOD COVERED Final Report
7. AUTHOR(s) Mr. Horst Gigerenzer Mr. Gary Strempek		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Fiber Materials, Inc. Biddeford, Maine 04005		8. CONTRACT OR GRANT NUMBER(s) DAAG46-77-C-0036 <i>new</i>
11. CONTROLLING OFFICE NAME AND ADDRESS Army Materials and Mechanics Research Center Watertown, Massachusetts 02172		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: IT163102D471 AMCMS Code: 613102.0710012 Agency Accession: DA OF4782
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE March 1978
		13. NUMBER OF PAGES
		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Graphite-aluminum composites Fabrication Pultrusion process		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) See reverse side		

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ABSTRACT

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The objective of this program was to establish the feasibility of producing sound rectangular bars by the pultrusion process. Previous work conducted under contract DAAG-46-76-C-0068 identified process problems for non-round sections. Addressed in the present work were:

- (a) Elimination of rectangular bar twisting during pultrusion;
- (b) Surface improvement by application of thin aluminum claddings;
- (c) Incorporation of thin titanium interleaf to the composite.
- (d) Further evaluations of composite heatreatability without cracking.

Rectangular bars of improved geometric uniformity, clad with aluminum and optionally interleaved with titanium were successfully fabricated by pultrusion processing. Twelve bars were delivered to AMMRC for evaluation. It was concluded that the pultrusion technology developed thus far has potential for the future fabrication of clad and/or interleaved, uniaxially graphite fiber reinforced aluminum-matrix, composite structural members.

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FOREWORD

This work is being performed by Fiber Materials, Inc. Biddeford, Maine for the Army Materials and Mechanics Research Center, Watertown, Massachusetts under Contract DAAG-46-77-C-0036. The Army Technical Supervisor is Mr. Jacob Greenspan. The FMI Program Manager is Mr. Horst Gigerenzer with Mr. Gary C. Strempek assisting as Project Engineer.

1.0 OBJECTIVES

In previous work conducted under Contract DAAG-46-76-C-0068, a new method of fabricating rod and bar forms of aluminum matrix graphite fiber composite was developed. This method consisted of consolidating an assembly of primary wire forms by warm drawing or pultruding this assembly through a constricted opening. The rods produced in this way exhibited exceptionally good geometric uniformity and good tensile strength. Rectangular bars, however, became twisted and warped. In addition, cracking occurred during heat treatments designed to strengthen the matrix.

The present work had the objective to establish the feasibility of producing sound rectangular bars by the pultrusion process. For this purpose the ensuing objectives were to incorporate modifications which would:

- (a) Eliminate twisting and warping previously experienced in rectangular sections.
- (b) Apply thin claddings of aluminum for surface improvement.
- (c) Bond a thin interleaf of titanium in the composite during fabrication.

An additional objective was to develop a heat treatment process to strengthen the matrix without introducing quench cracks.

2.0 SUMMARY

The pultrusion or drawing process for consolidating an assembly of aluminum graphite wires, previously developed under Contract DAAG-46-76-C-0068, was modified in accordance with the above objectives. Rectangular bars of improved geometric uniformity, clad with thin aluminum, and optionally interleaved with titanium were produced. After optimization of process parameters, 12 bars were fabricated and delivered to AMMRC as required.

The problem of twisting and distortion of the bar was overcome by incorporating a sliding (shear) interface in the billet lay-up. This consisted of oxidized titanium

foil coated with a layer of boron nitride wash, providing a mechanism to break continuity of stress transfer between the billet containment and the graphite-aluminum composite, eliminating distortion.

Additional billet modifications were accomplished to achieve a more uniform aluminum cladding of the composite bars and to allow incorporation of a thin titanium interleaf, to demonstrate pultrusion fabrication of graphite-aluminum-titanium laminate structures. Some corrugation of the titanium interleaf occurred and little improvement in transverse strength was observed. This may relate to weak Ti-Al bonding, but requires further analysis.

Tensile testing of rectangular section bars indicated apparent lower tensile strength translations than previously observed for round section bar stock. However, comparison of results from flat and round section bars indicated that the tensile strengths were a function of test specimen configuration. The highest tensile strength observed in this work for rectangular bars was 104,000 psi, representing 62% translation from wire preform (168 ksi average preform strength) to final bar. When a round specimen was tested, obtained from a round section bar fabricated under the same process conditions, a tensile strength of 124,000 psi (72% translation of 185 ksi average preform strength) was observed, while a rectangular specimen from the same bar yielded a UTS of 95,000 psi (56% translation). The round specimen result was equivalent to previous results obtained for round bar stock fabricated under contract DAAG-46-76-C-0068 where 73% translation was typical. From these observations it appears that equivalent composite consolidation levels were achieved for rectangular bars and that the apparent lower tensile strengths are due to test specimen configuration, rather than the fabrication process.

A limited investigation of heat treatment (solution, quench, and age) on rectangular section bars was also conducted to determine the feasibility of such heat

treatments to increase transverse strengths. Several quench methods, minimizing quench rate from the solutionizing temperature, were investigated to eliminate cracking of the composite bar experienced previously. Results of these attempts showed that cracking of the composite persisted, using standard heat treating methods. Therefore, effect of heat treatment on transverse strength could not be evaluated.

3.0 INTRODUCTION

3.1 Background

Fabrication and thermal-mechanical working of uniaxial reinforced graphite-aluminum round bar sections via the pultrusion (hot-drawing) process with accompanying high strengths and elastic moduli was successfully demonstrated under contract No. DAAG-46-76-C-0068⁽¹⁾. This previous work resulted in attaining tensile strengths in excess of 100,000 psi and 16-20 msi moduli for pultruded T300 G/6061 Al and T300 G/356 Al composite round bars. A 72% strength translation (from composite wire to fabricated bar) was observed for round bars. Rectangular bars were also fabricated by pultrusion, but exhibited a twist along the axis of the bar. Attempts to straighten one bar after pultrusion, by hot press forming partially corrected this condition; however, micro cracks were evident upon examination of the bar ends. Heat treatment (solution, quench, and aging) of rectangular bars resulted in gross splitting during quench cycle. Due to these process difficulties, suitable specimens from rectangular bars were not obtained for mechanical testing.

The work dealt with in this report is a continuing effort at developing the hot-pultrusion process for fabricating simple graphite aluminum structural shapes and addresses previous process problems. Particularly the aim was to eliminate axial distortion (twisting) in rectangular graphite-aluminum bars, through modification of the composite billet lay-up and pultrusion hardware (if required). Paralleling

(1) Fabrication of Discontinuous Graphite-Aluminum Composites Via Pultrusion, February, 1977; Final Report, Contract No. DAAG-46-76-C-0068; Army Materials and Mechanics Research Center, Watertown, Massachusetts.

these efforts were additional process improvements in billet lay-up to allow application of a uniform cladding and the introduction of a titanium interleaf to demonstrate pultrusion of graphite-aluminum-titanium laminates. Heat treatment of graphite-aluminum composite bars was further investigated. Modifications were evaluated in terms of feasibility and overall fabrication process improvements whenever results permitted.

3.2 Technical Approach

The first objective was to eliminate axial twisting of rectangular bars during pultrusion fabrication. Several process related factors were considered the cause. These were:

- (a) Misalignment of pulling ram and die assembly.
- (b) Frictional drag between die and billet.
- (c) Stress interactions between dissimilar materials within billet assembly (i.e. composite anisotropy and thermal expansion mismatches).

These factors acting, singularly or in combination, were thought to cause bar distortion. Corrective actions considered were:

- (1) Improve alignment of ram and die in relation to one another.
- (2) Use improved high temperature/pressure lubricants.
- (3) Introduce sliding layer in billet lay-up (eliminate stress interactions).
- (4) Impose external forming constraints to billet.

A schematic representation of the pultrusion apparatus (the same set-up as used in previous work) is shown in Figure 1. Preliminary analysis and experiments showed that realignment of die and ram as well as use of improved pultrusion lubricants had little effect on axial distortion. External constraints were considered but this would require excessive modifications and cost.

The most feasible approach appeared to be the incorporation of a shear interface to provide a mechanism for the relief of stresses responsible for the distortion. Various interlayers were considered ranging from internal lubricants to

SECTIONAL VIEW OF HOT-DRAWING PROCESS SET-UP

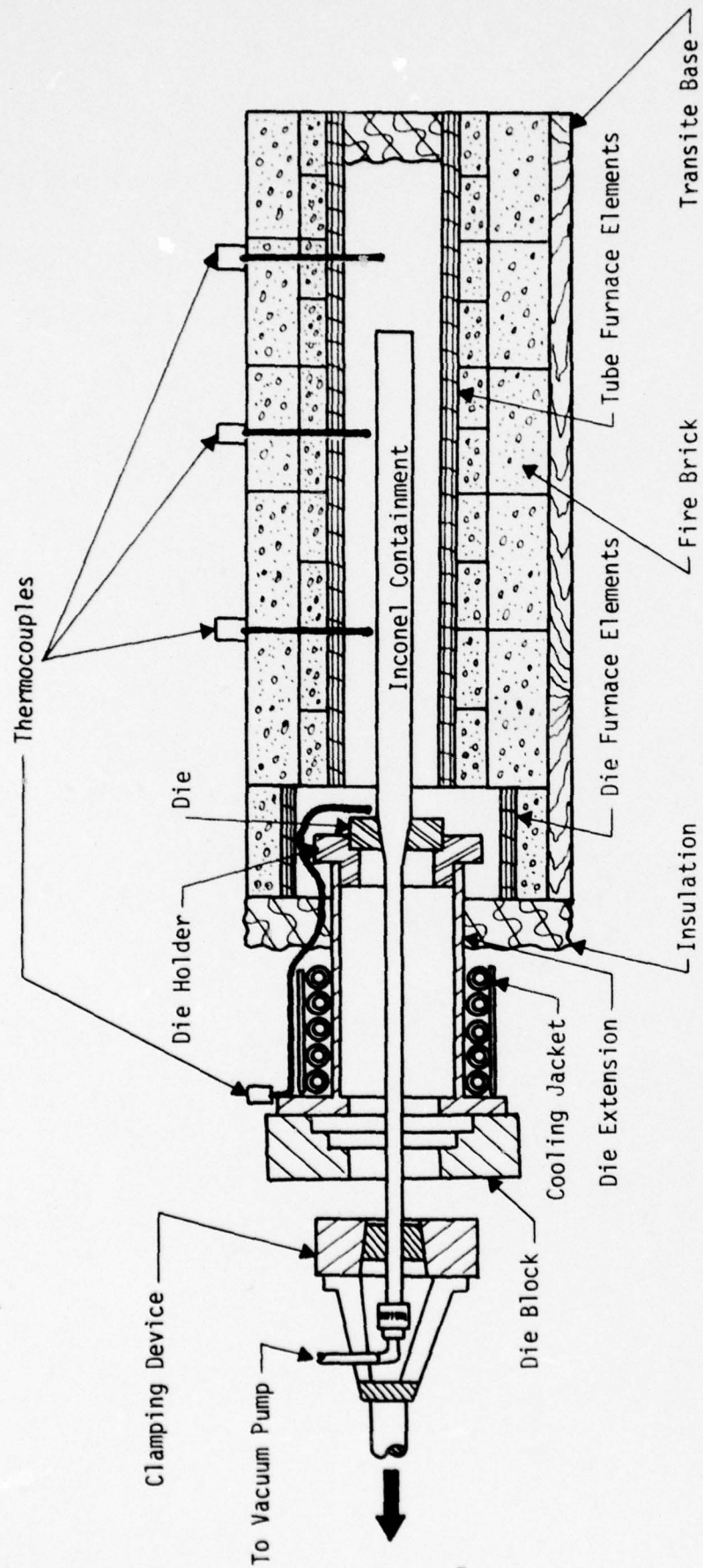


Figure 1

sliding foil layers and combinations thereof which could (a) effectively reduce stress transfer between composite and containment, (b) be easily incorporated into existing billet lay-up and (c) remain unbonded to the composite bar. This approach prevented bar distortion.

To incorporate cladding and interleaf components in the bar, the approach was to introduce the appropriate components into the billet and to modify pultrusion parameters as needed. Previous difficulties during heat treatment were approached by investigating reduction of quench rate from solutionizing temperature.

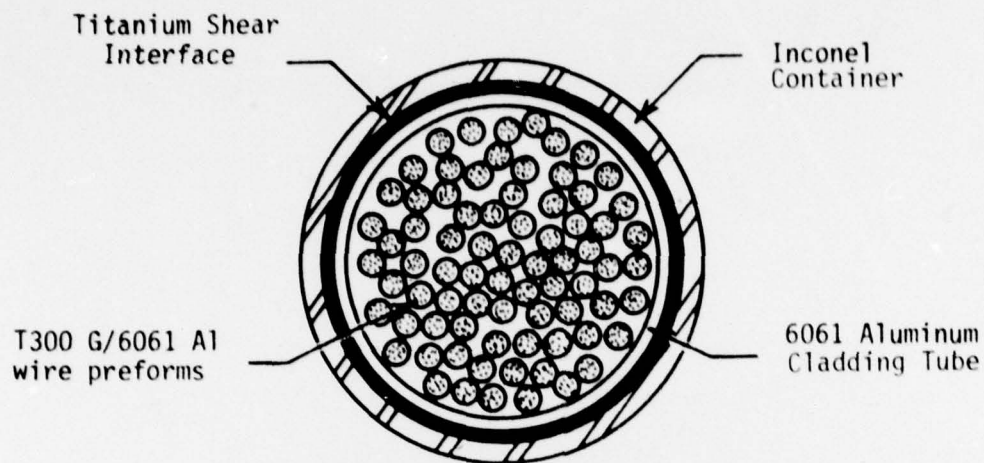
4.0 PROCESS MODIFICATIONS FOR PULTRUSION OF RECTANGULAR GRAPHITE-ALUMINUM BARS

4.1 Addition of Shear Interface

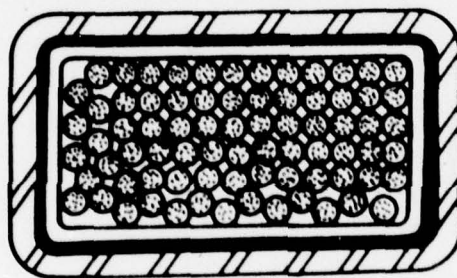
To eliminate axial twisting during pultrusion of rectangular graphite-aluminum bars, a sliding (shear) interface was introduced between the composite lay-up and the billet container (Figure 2). The interface consisted of 0.001" thick titanium foil, cut to fit and completely surround, the inside surfaces of the Inconel container. The foil was oxidized, with the face adjacent to the composite lay-up coated with boron nitride. The oxidized surfaces prevent sticking while the boron nitride coating acted as a solid lubricant. A bake-out (500°F/30 minutes) of the coated foil in inert atmosphere was performed to eliminate volatiles and cure the coating prior to insertion of the foil into the billet lay-up.

4.2 Addition of Aluminum Cladding

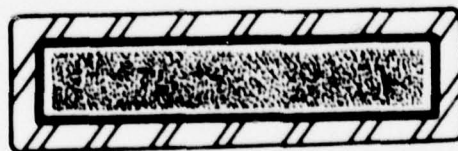
The 6061 aluminum cladding was applied by insertion of a thin walled tube between the titanium foil and the graphite-aluminum wire lay-up (Figure 2). During pultrusion the tube was deformed and bonded to the graphite-aluminum composite resulting in a uniform outer cladding. The cladding applied was of sufficient thickness (~0.028") to allow clean-up by machining and still maintain a cladding thickness in the range of 0.010" to 0.020".



(a) Initial Lay-Up (loosely packed)



(b) Pre-formed Billet (tightly packed)



(c) Pultruded Assembly
(Consolidated G/Al composite)

FIGURE 2

CROSS SECTION OF RECTANGULAR GRAPHITE-ALUMINUM BILLET

AT VARIOUS STAGES OF PROCESSING

4.3 Addition of Titanium Interleaf

Additional modifications to the billet lay-up were required to bond a titanium interleaf within the graphite-aluminum structure resulting in a graphite-aluminum-titanium laminate. Wire preforms were divided in two bundles and wrapped with a layer of 0.002" thick 6061 aluminum foil. A 0.001" thick pure titanium interleaf, placed between the wire bundles, was wrapped in aluminum foil and inserted into a 6061 aluminum cladding tube (Figure 3). The titanium interleaf and mating aluminum surfaces were thoroughly cleaned to remove oxides and contaminants. Aluminum surfaces were abraded and ultrasonically degreased in acetone. Titanium foils were acid etched in 10 ml HF, 45 ml HNO₃ and 45 ml H₂O followed by a cold water rinse and dried with warm air. This cleaning operation was performed immediately before lay-up. The assembly was inserted into the Inconel container with titanium shear layers in place, sealed, and vacuum leak checked.

4.4 Preforming of the Composite Billet

The assembled and sealed composite billet was preformed by cold-drawing through a sizing die (0.850" X 0.500"). The preforming reduction packed the contents of the billet, locking the composite wires in place to minimize wire slippage and crossover during hot-pultrusion. Previous pultrusion trials indicated that a one step reduction from initial billet to final rectangular bar size, resulted in excessive fiber breakage and misalignment, lowering mechanical properties of the composite bar. By minimizing breakage and misalignment a more uniform composite structure should also result.

In addition preforming should extend die life due to reduced wear of the tungsten carbide insert. One-step pultrusion of the composite billet through the final die was found to impart high stress concentrations at die corners which led to cracking and subsequent failure of the tungsten carbide insert.

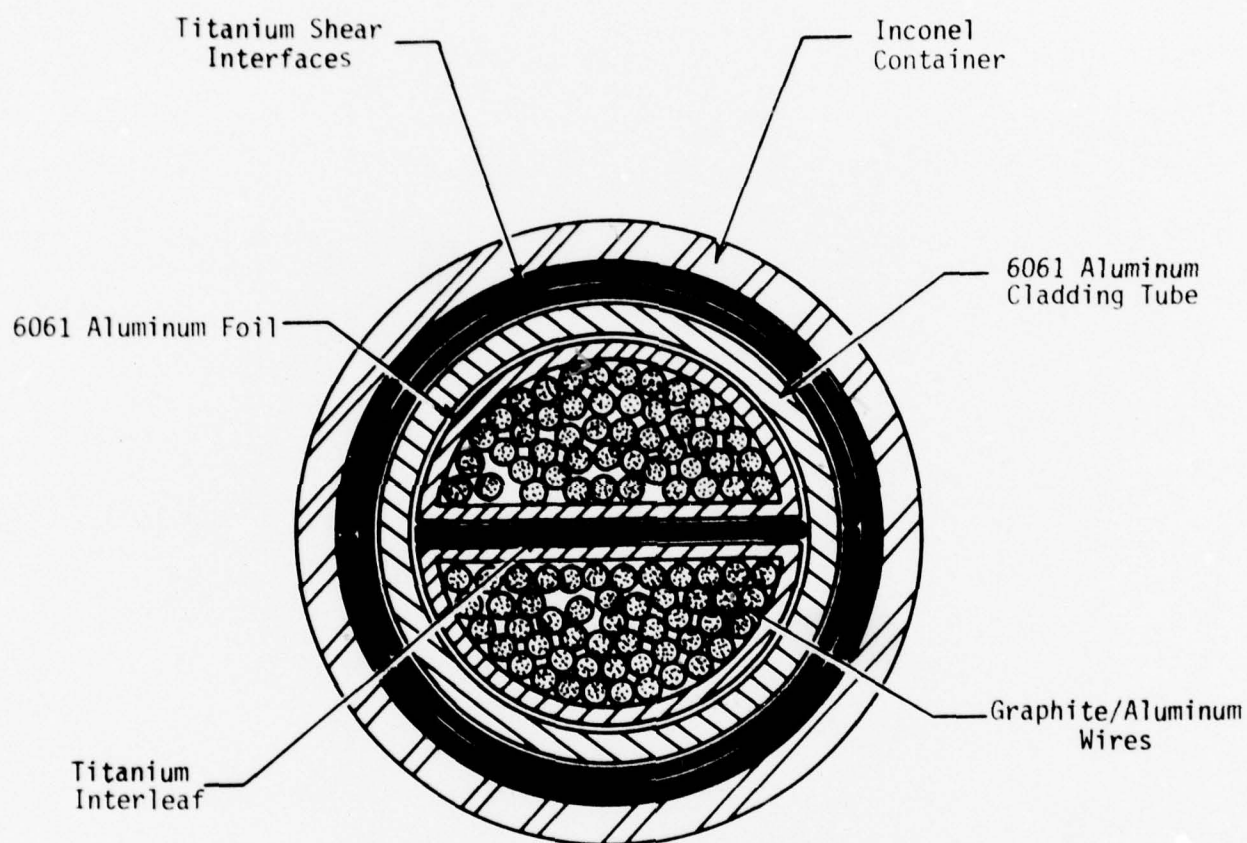


FIGURE 3
CROSS SECTION OF RECTANGULAR GRAPHITE-ALUMINUM-
TITANIUM BILLET ASSEMBLY

4.5 Process Parameters

A pultrusion process temperature of 1050⁰F (30⁰ below the solidus for 6061 aluminum) was chosen based on diffusion bonding processing temperature profiles for hot pressing of 6061 aluminum matrix graphite-fiber composites. This change in processing temperature from that used in previous round bar fabrication (950⁰F) served to minimize stress build-up within the billet during pultrusion while still processing in the solidus range. Consolidation occurred under vacuum (< 0.1 Torr) at a pultrusion rate of 2.5 inches/second through a 0.25" x 0.85" die. Based on the calculated cross-sectional area of about 0.15 square inches of preform material the effective reduction in area was about 5%. These conditions, with the exception of processing temperature, were equivalent to those used previously to fabricate round section bars. Nominal dimensions of the rectangular bars were 0.2" x 0.7" x 12".

5.0 RESULTS AND DISCUSSION

5.1 Preliminary Processing Trials

Several preliminary pultrusion trials using available graphite-aluminum wire preforms were performed (Table 1). These trials were conducted to determine the effect on axial twisting of various shear interface lay-ups (foils and coatings) between the billet containment and the composite. The use of graphite foil as a shear interface in an early attempt (Trial 213), showed the effect of introducing a stress discontinuity between container and composite. Although severe fragmentation of the composite bar occurred as a result of high reduction (>15% RA), a flat non-twisted section resulted (Figure 4). When unreinforced aluminum wires were pultruded (Trial 214) without a foil interface, no axial twisting occurred. This result indicated that axial twisting is a strong function of inherent composite anisotropy due to uniaxial fiber reinforcement. Consideration of thermal expansion mismatch between the Inconel container and the graphite-aluminum composite

TABLE 1
KEY PROCESSING TRIALS FOR PULTRUSION OF RECTANGULAR GRAPHITE-ALUMINUM BARS

<u>Trial</u>	<u>Shear Interface</u>	<u>Temperature (°F)</u>	<u>Result</u>
194	Boron Nitride	1140	Axial twisting
213	0.005" Grafoil	1050	No axial twist, fragmentation of composite bar
214(a)	Boron Nitride	1050	No axial twist
216	0.001" Oxidized Titanium Foil	1000	No axial twist, surface wrinkles
218	0.001" Oxidized Inconel 600 Foil	1000	No axial twist, no improvements in surface finish
227	0.001" Oxidized Titanium Foil (2 layers, one with Boron Nitride Coating)	950	No axial twist, improved surface finish
232	0.001" Oxidized Titanium Foil (2 layers, one with Boron Nitride Coating)	1050	No axial twist, some edge wrinkling

NOTE:

(a) Lay-up consisted of aluminum welding wire (4043) only.

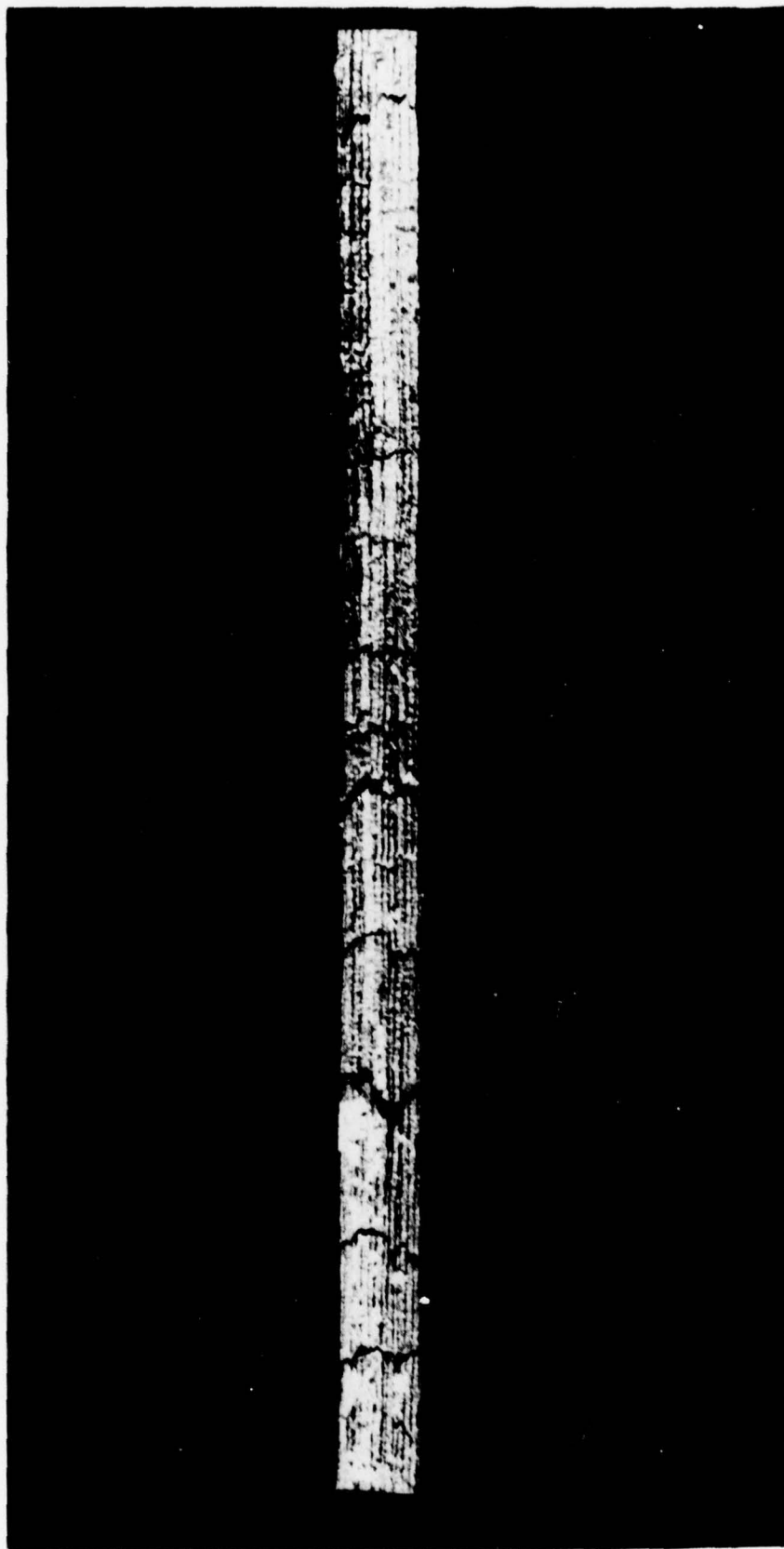


FIGURE 4

GRAPHITE-ALUMINUM PULTRUDED FLAT BAR SEVERLY FRACTURED ($> 15\%$ R.A.)

was also thought to contribute towards the overall distortion of the billet. From these trial and error results it became evident that elimination of stress interactions between the billet materials was necessary. Subsequent substitution of titanium foil layers to act as a shear interface and break continuity of stress transfer resulted in elimination of axial twisting and improvements in surface finish (Figure 5). Further trials with various foil configurations resulted in process refinements and consistency.

5.2 Metallographic Examinations

Composite wire preforms fabricated by the Ti/B liquid metal infiltration process showed some minor casting defects (gas holes and fiber rich areas). In general, however, the wire was of good quality and sound structure (Figure 6A). Examination of metallographic sections from consolidated graphite-aluminum rectangular bars (nominal dimensions 0.2" X 0.7" 12") indicated that sound composite structures were achieved by pultrusion processing in the solid state (Figure 6B). Pultruded bars showed good consolidation and fewer defects than observed in the wire preforms. This was attributed to healing of defects during pultrusion processing.

Introduction of a titanium interleaf into the composite bar during pultrusion was successfully demonstrated. Good bonding was obtained when acid etching of the titanium to remove oxides was performed, prior to lay-up. It was necessary to include a layer of 6061 aluminum foil between the composite and titanium interleaf to obtain uniform bonding over the entire titanium surface (Figure 7B). Early fabrication trials without the aluminum interface showed areas of poor bonding where the graphite fibers contacted the titanium interleaf. Rippling of the titanium interleaf (Figure 7A) as well as small fractures within the interleaf cross section were observed in several metallographic sections.

5.3 Tensile Properties

Tensile tests in the longitudinal (fiber) direction were performed on

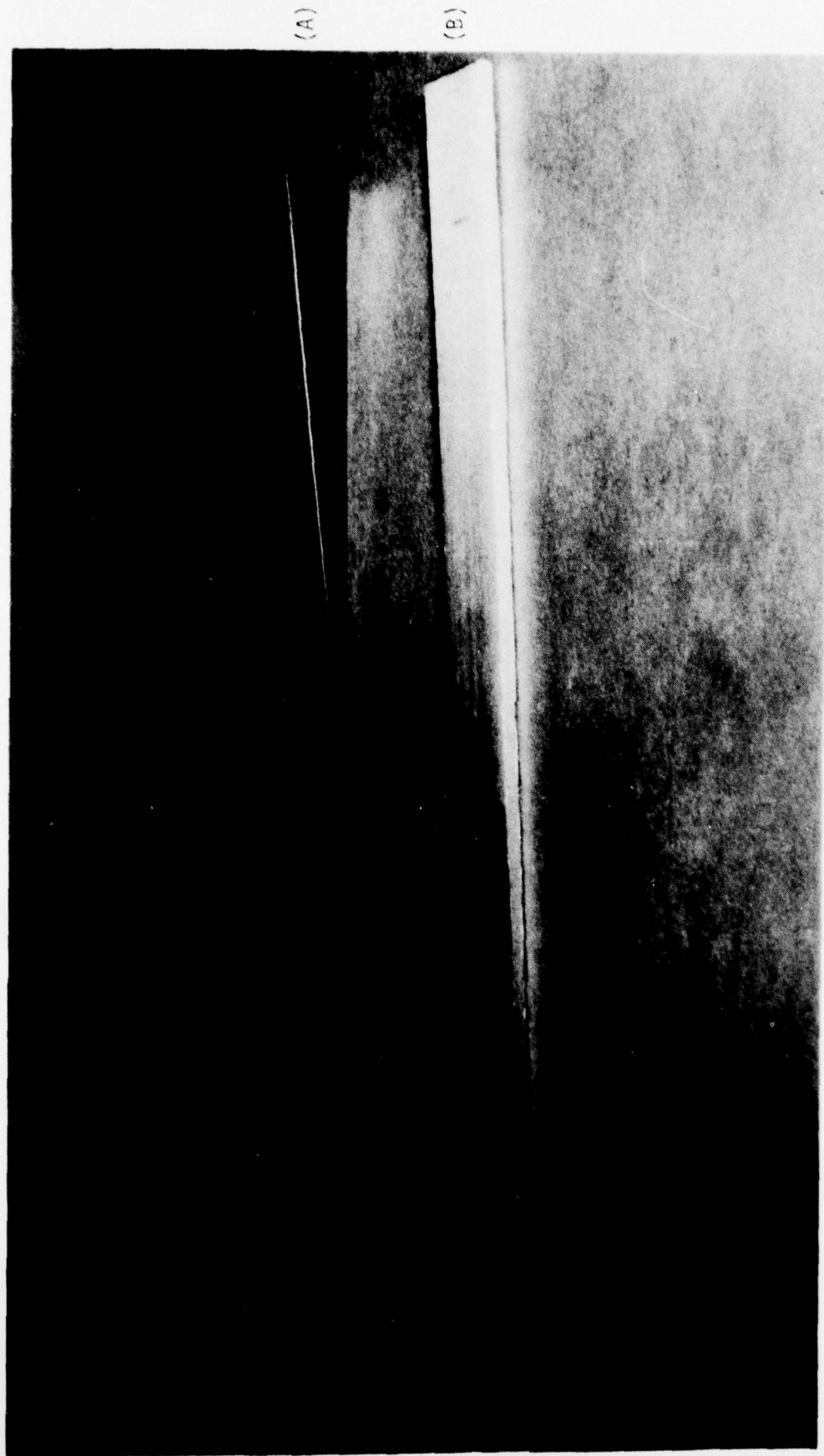
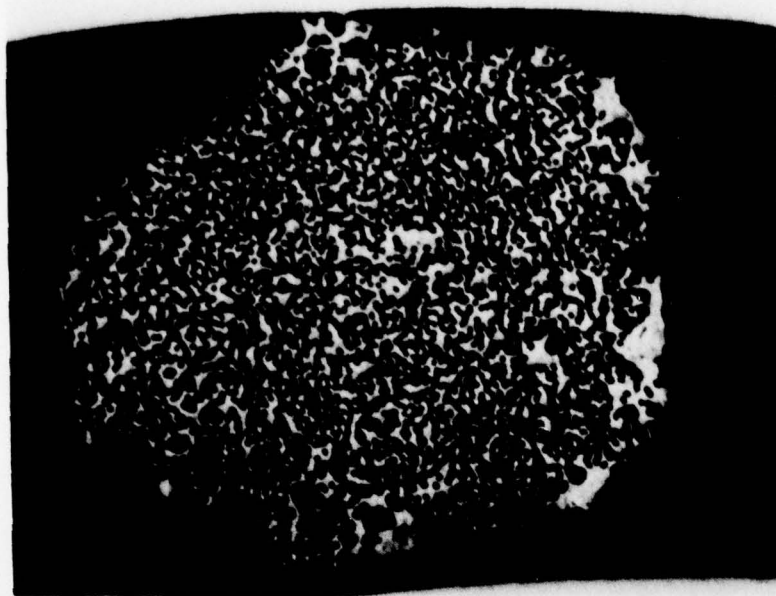
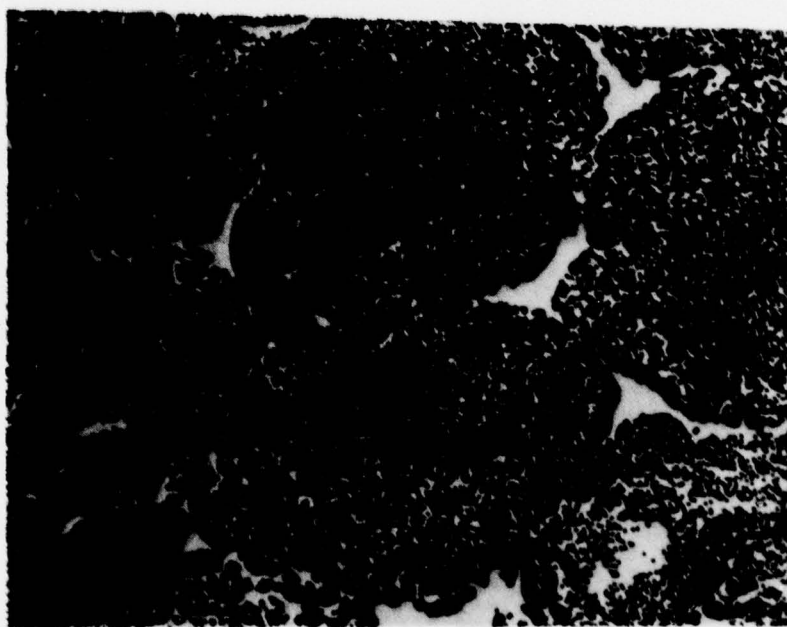


FIGURE 5
GRAPHITE-ALUMINUM PULTRUDED BARS WITH TWIST (A) AND WITHOUT
TWIST (B) DUE TO INCORPORATION OF SHEAR INTERFACE



(A) Wire Preform

Mag. 100x
As-Polished



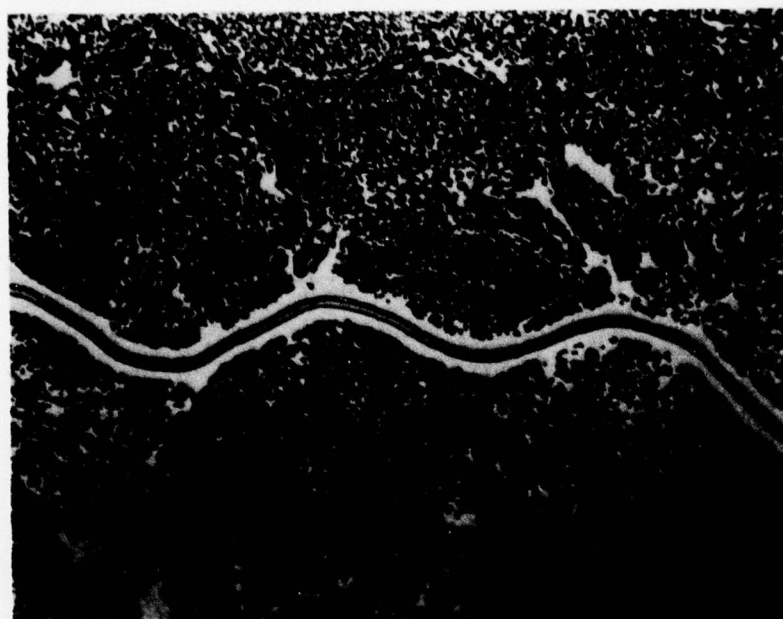
(B) Pultruded Rectangular Bar

Mag. 65x
As-Polished

FIGURE 6

TYPICAL TRANSVERSE MICROSTRUCTURE OF T300

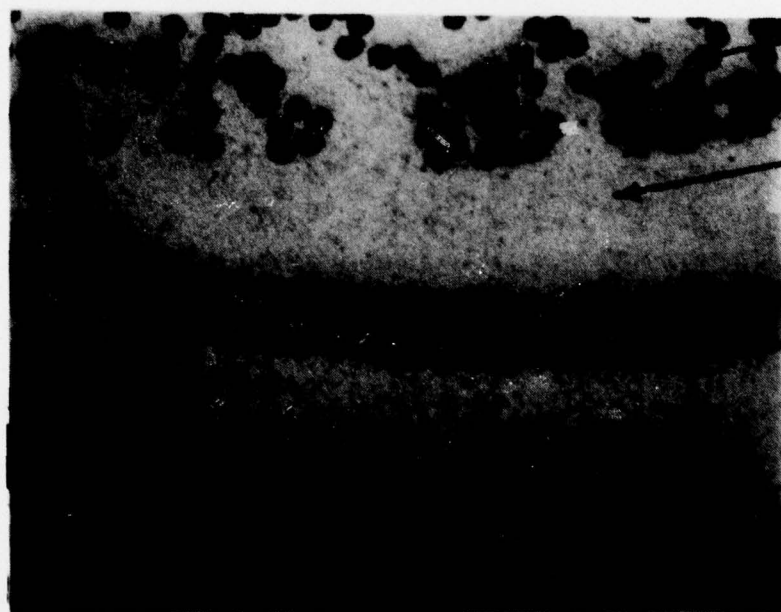
GRAPHITE-6061 ALUMINUM COMPOSITE



T300 G/6061 Al

(A)

Mag. 50x
As-Polished



T300 G/6061 Al

(B)

Mag. 500x
As-Polished

FIGURE 7

TYPICAL TRANSVERSE MICROSTRUCTURE OF A PULTRUDED
GRAPHITE-ALUMINUM RECTANGULAR BAR WITH TITANIUM INTERLEAF

rectangular section T300 graphite-6061 aluminum bars and strength translations were compared to results previously obtained for round bars⁽¹⁾. Table 2 summarizes these comparisons.

Tensile strengths as high as 104,000 psi were observed for flat tensile specimens machined from rectangular section composite bars (bar #235). Flat tensile specimens were machined in accordance with Figure 8. An apparent lower translation of tensile strength from the wire preform to pultruded rectangular section bars, than previously observed for pultruded round section bars, was shown to be due to tensile specimen design. Flat tensile specimens machined from both round and rectangular section pultruded T300 G/6061 Al composite bars showed similar strength translations (56 and 57% respectively). Round tensile specimens (Figure 9) taken from the same bar as the flat specimens, yielded translations in the range of 69% (24% increase). The marked effect of specimen design is probably due to low transverse strength in G/Al. Examination of fracture faces showed that specimens are not failing in pure tension but that failure is induced by shear, a result of low composite transverse strength. The degree to which shear initiated failure affects testing apparently is a function of specimen design. Figure 10 shows typical stress-strain behavior for both T300 G/6061 Al wire preform and pultruded rectangular section bars.

Transverse tensile results obtained from rectangular coupons were characteristically low for graphite-aluminum. Similar specimens taken from graphite-aluminum reinforced with one titanium interlayer showed no considerable improvement (Table 3). Substantial improvements in transverse strength were not expected for the titanium reinforced laminate consisting of one layer of titanium foil. The

(1) Fabrication of Discontinuous Graphite-Aluminum Composites Via Pultrusion, February, 1977; Final Report, Contract No. DAAG-46-76-C-0068; Army Materials and Mechanics Research Center, Watertown, Massachusetts.

TABLE 2

MECHANICAL PROPERTIES OF PULTRUDED T300 GRAPHITE/6061 ALUMINUM BARS

(40 v/o Fiber)

Bar	Processing Temperature (°F)	Bar Configuration	Specimen Configuration	Elastic Modulus		Ultimate Tensile Strength (ksi)	Strain (%)	Percent UTS Translation (%)
				$E_1 \times 10^6$ (psi)	$E_2 \times 10^6$ (psi)			
147*	940	Round	Round	20.3	14.6	134	0.89	72
			Round	15.5	14.3	130	0.90	70
230	1040	Round	Round	17.7	15.2	124	0.80	73
			Round	17.2	----	109	0.67	64
			Rectangular	17.9	14.0	95	0.67	56
235	1040	Rectangular	Rectangular	16.2	12.0	89	0.71	52
			Rectangular	17.6	14.3	104	0.72	62

* Result from Reference 1

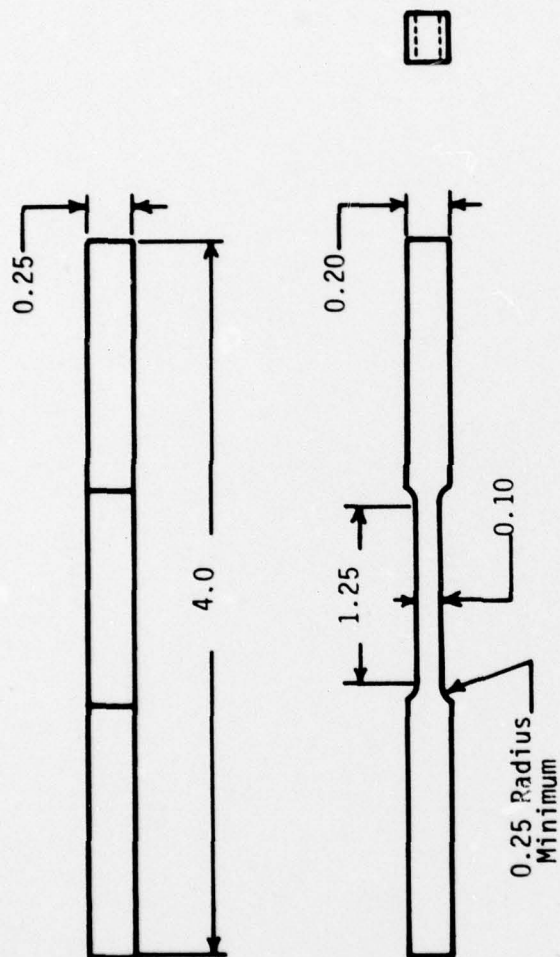


FIGURE 8

RECTANGULAR TENSILE SPECIMEN CONFIGURATION

ROUND TENSILE SPECIMEN CONFIGURATION

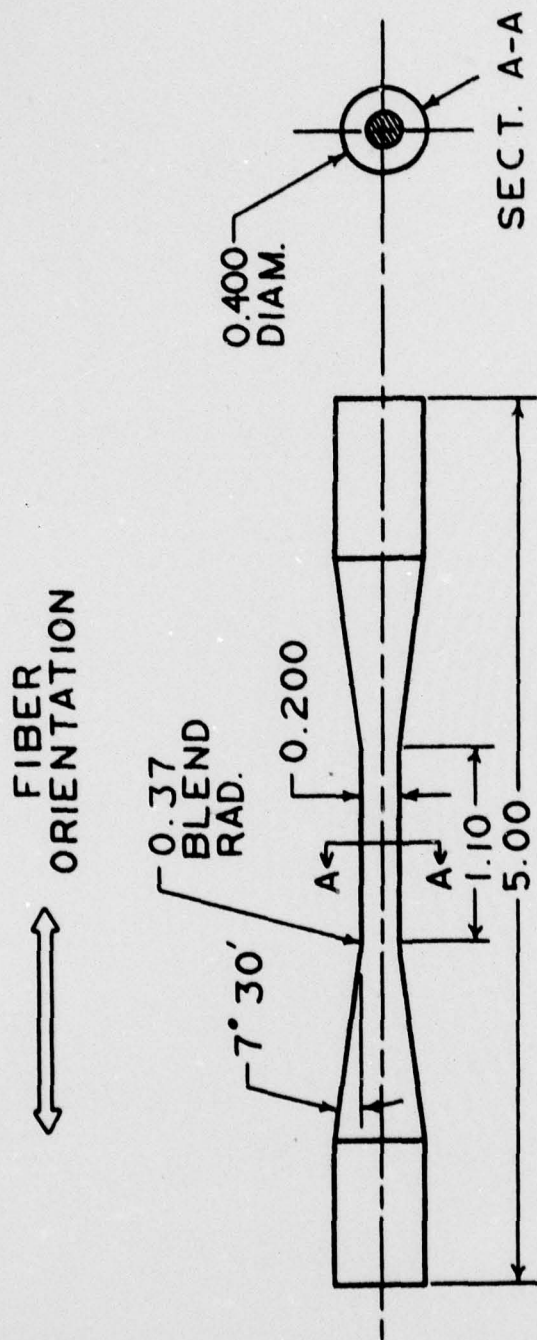


Figure 9

FIGURE 10
TYPICAL STRESS-STRAIN BEHAVIOR OF T300 G/6061 AL COMPOSITE
(40 v/o)

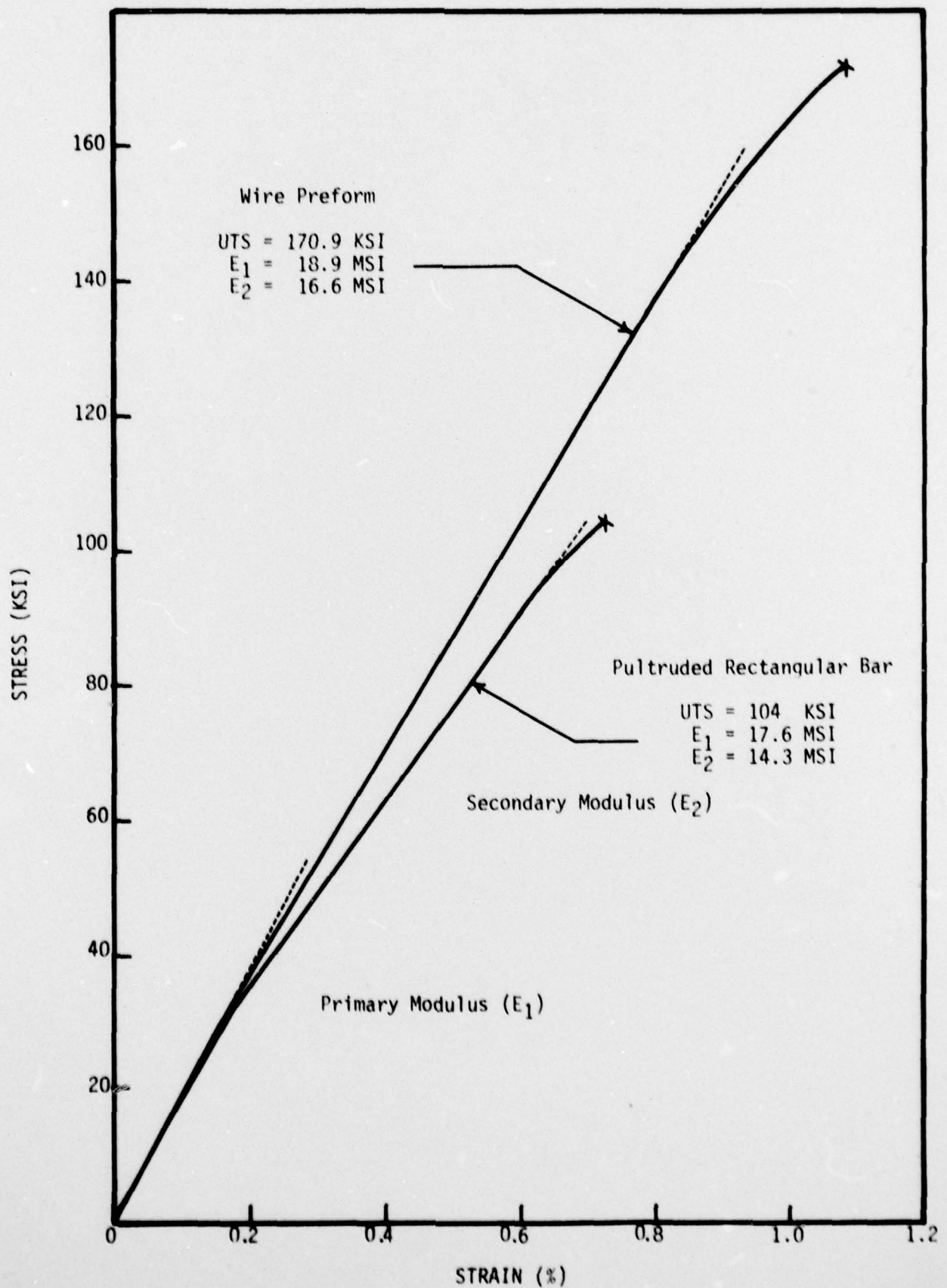


Table 3
TRANSVERSE TENSILE STRENGTH RESULTS OF AS-PULTRUDED BARS

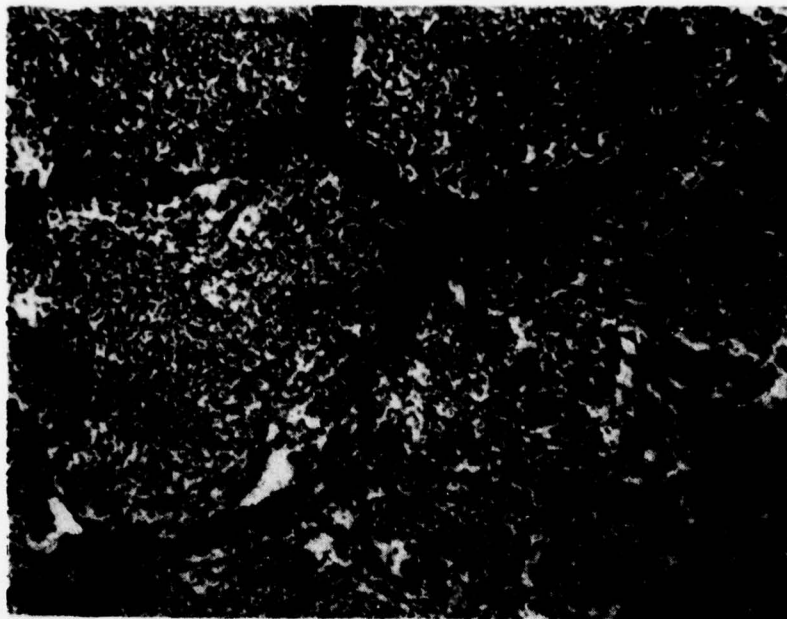
<u>Bar</u>	<u>Material</u>	UTS (psi)
235	G-Al	1090 1400
236	G-Al-Ti	1200 1630

reinforcement was included in the bar only for purposes of fabrication process demonstration. It appears feasible that multileaf Ti lay-ups could be fabricated with resultant improvements in composite transverse strength.

5.4 Heat Treatment

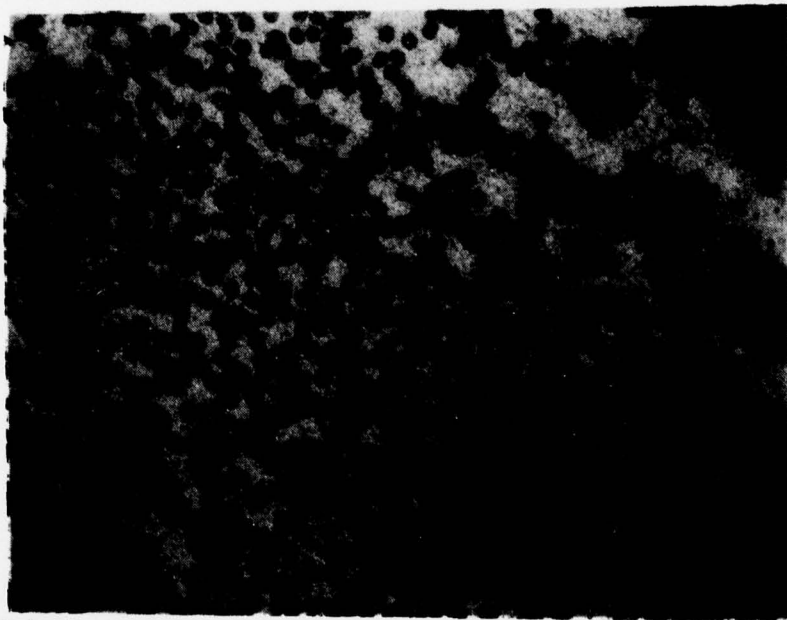
Previous attempts at heat treating rectangular section graphite-aluminum bars resulted in cracking during quenching into brine from the solutionizing temperature (1000°F). In general a fast quench rate is desirable to achieve optimum matrix response on subsequent aging of the composite. However, cracking occurred, due to thermal expansion mismatch between the graphite fibers and aluminum alloy matrix. The severity of cracking varies from micro-cracks to macro-cracks (Figure 11).

Further limited attempts under this contract were investigated to heat treat the composite matrix. Both water and air spray techniques were employed to reduce quench rates. Table 4 summarizes results of these attempts. Samples on which the pultrusion container (Inconel cladding) was not removed appeared to be least susceptible to cracking when quenched by air spray from the solutionizing temperature. However, improvements in composite matrix properties would not be



(A) Macro-Cracks Along Wire Boundaries

Mag. 65x
As-Polished



(B) Micro-Cracks Within Wire Boundaries

Mag. 310x
As-Polished

FIGURE 11

TYPICAL TRANSVERSE MICROSTRUCTURE SHOWING CRACKS WHICH
OCCURRED UPON QUENCHING FROM SOLUTIONIZING TEMPERATURE

TABLE 4

HEAT TREATMENT TRIALS ON RECTANGULAR SECTION T300 G/6061 AL

<u>Trial</u>	<u>Cladding</u>	<u>Quench (from 1000°F)</u>	<u>Result</u>
1	none	Water Spray	Cracking
2	none	Air Blast	Cracking
3	Inconel	Water Spray	Cracking
4	Inconel	Air Blast	No cracking upon visual examination

expected under these low quench rates with subsequent aging. It was concluded that conventional solution, quench and aging treatments for rectangular section graphite-aluminum composites is not readily feasible. However, non-conventional heat treatments (ex., quenching under pressure followed by aging) may allow improvements and should be further addressed.

6.0 CONCLUSIONS

1. Introduction of a sliding (shear) interface to the graphite-aluminum pultrusion billet lay-up is an effective means whereby axial distortion, previously experienced, is eliminated during pultrusion of rectangular section bars from uniaxial wire preforms.
2. Uniform claddings and potentially reinforcing titanium interleafs can be incorporated during pultrusion fabrication of simple rectangular section graphite-aluminum bars.
3. Excellent consolidation and an equivalent degree of strength translation (from wire to bar) occurs for pultruded rectangular sections, comparable to round section bars previously fabricated via pultrusion.
4. The pultrusion process technology for uniaxial reinforced graphite-aluminum composites has advanced to allow routine fabrication of simple structural shapes (round and rectangular sections). More complex sections (angles) appear feasible with minor modifications in billet lay-up and pultrusion die designs.

7.0 RECOMMENDATIONS

1. Transverse strength improvements for graphite-aluminum structures may be achieved by claddings, reinforcing interleafs and/or heat treatment of composite matrix. Limited effect of heat treatment was shown previously for round section bars while the feasibility of applying claddings and

incorporating potentially reinforcing interleaves during fabrication, has been demonstrated in this work. Therefore, it is recommended that follow-on efforts should emphasize full development of these approaches to achieve significant transverse strength improvements for graphite-aluminum pultruded structural sections.

2. Apply developed pultrusion process technology to demonstrate the fabrication of aluminum clad complex graphite-aluminum angle structural sections (i.e., tees, zees, etc.).